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SPECIMEN HETEROGENEITY ANALYSIS; A PRIMER (PREPRINT)

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UES, Inc.

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14. ABSTRACT
The characterization and the quantification of specimen heterogeneity is an issue that is intimately related to the precision, or variability, of the x-ray measurements that are made on a specimen. While the precision of electron probe micro-analysis (EPMA) techniques has been studied thoroughly over the past 55 years, it is less often discussed in relation to the topic of specimen heterogeneity. For the beginning analyst, the relationship between the statistical interpretation of the data and the application of the various heterogeneity equations can be confusing. The NIST-Analysis of Variance (ANOVA) procedure provides a rigorous method for evaluating heterogeneity within research materials; however, a quick estimate of the heterogeneity range is often all that is required for a single specimen that is not intended to be sued as a standard reference material [1]. For these instances, legacy equations, such as the one proposed by Goldstein, et al, have been suggested because they are quick and easy to apply [2].

15. SUBJECT TERMS
Electron probe micro-analysis, EPMA, specimen heterogeneity, ANOVA, legacy equation

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Specimen Heterogeneity Analysis ; A Primer

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The characterization and the quantification of specimen heterogeneity is an issue that is intimately related to the precision, or variability, of the x-ray measurements that are made on a specimen. While the precision of electron probe micro-analysis (EPMA) techniques has been studied thoroughly over the past 55 years, it is less often discussed in relation to the topic of specimen heterogeneity. For the beginning analyst, the relationship between the statistical interpretation of the data and the application of the various heterogeneity equations can be confusing. The NIST-Analysis of Variance (ANOVA) procedure provides a rigorous method for evaluating heterogeneity within research materials; however, a quick estimate of the heterogeneity range is often all that is required for a single specimen that is not intended to be used as a standard reference material [1]. For these instances, legacy equations, such as the one proposed by Goldstein, et al, have been suggested because they are quick and easy to apply [2]. The present work takes a close look at the equation proposed by Goldstein et al, and suggests that the equation should be revised to make it better able to describe the homogeneity range found within a single specimen. The study will also include modifications to the equation for the standard deviation of the mean concentration that was proposed by Lifshin, et al [3]. Though the Lifshin, et al equation was not originally intended for use in heterogeneity studies, it can be generalized, and thus made applicable for this purpose.

Multiple EPMA-WDS heterogeneity analyses were conducted on two different titanium alloy specimens; one a nominally homogeneous alloy (Alloy 1), the other a non-homogeneous alloy (Alloy 2). Following the procedures outlined in Marinenko, et al, a full ANOVA heterogeneity study was done on both of the specimen materials [1]. The ANOVA study was used to establish benchmarks for the evaluation of two equation revisions that are proposed in this work. The large amount of data collected for the ANOVA study also made it possible to directly calculate the variation within the weight percent data set, providing a second benchmark for comparisons. Tables 1-2 (Alloy 1) and Tables 3-4 (Alloy 2) provide a summary of the calculation results of these heterogeneity studies. The homogeneity range for each alloy is given in Table 1 and Table 3, with the corresponding homogeneity levels being shown in Tables 2 and Table 4, respectively. The revised Goldstein, et al and the Lifshin, et al equations perform well in comparison with the two benchmark studies.

References

[1] Marinenko, R. and Leigh, S., Microscopy and Microanalysis, 10, #4, (2004), 491.
[2] Goldstein, J.I. et al, Scanning Electron Microscopy and X-ray Microanalysis, 2nd Edition, Plenum Press, NY,1992.
[3] Lifshin, E. et al, Microscopy and Microanalysis, 4, #6, (1998), 498.
[4] The authors would like to thank the following people for many helpful discussions related to the investigation of this topic; Dale Newbury and Ryna Marinenko of N.I.S.T., Eric Lifshin of

S.U.N.Y., Albany, Paul Shade, Robert Wheeler, and Michael Uchic of the Air Force Research Laboratory, and Dan Kremser of Battelle.

TABLE 1. Alloy 1 Homogeneity Range (3-Sigma Confidence)

	Al	Ti	Zr	Mo	Sn	
(Mean Concentration)	*(6.1)*	*(85.9)*	*(3.9)*	*(2.02)*	*(2.02)*	*(% wt.)*
Weight Percent Data Set	± 0.1	± 0.6	± 0.2	± 0.04	± 0.04	*(% wt.)*
Marinenko - ANOVA	± 0.1	± 0.7	± 0.2	± 0.05	± 0.04	*(% wt.)*
Goldstein - Original	± 0.01	± 0.04	± 0.01	± 0.002	± 0.002	*(% wt.)*
Goldstein - Revised	± 0.1	± 0.6	± 0.2	± 0.04	± 0.03	*(% wt.)*
Lifshin - Generalized	± 0.1	± 0.6	± 0.2	± 0.04	± 0.04	*(% wt.)*

TABLE 2. Alloy 1 Homogeneity Level (3-Sigma Confidence)

	Al	Ti	Zr	Mo	Sn	
(Mean Concentration)	*(6.1)*	*(85.9)*	*(3.9)*	*(2.02)*	*(2.02)*	*(% wt.)*
Weight Percent Data Set	2%	1%	5%	2%	2%	
Marinenko - ANOVA	2%	1%	5%	2%	2%	
Goldstein - Original	0.1%	0.04%	0.3%	0.1%	0.1%	
Goldstein - Revised	2%	1%	5%	2%	2%	
Lifshin - Generalized	2%	1%	5%	2%	2%	

TABLE 3. Alloy 2 Homogeneity Range (3-Sigma Confidence)

	Al	Ti	Mn	Nb	
(Mean Concentration)	*(31)*	*(53)*	*(3)*	*(13)*	*(% wt.)*
Weight Percent Data Set	± 5	± 4	± 1	± 1	*(% wt.)*
Marinenko - ANOVA	± 6	± 4	± 1	± 1	*(% wt.)*
Goldstein - Original	± 0.3	± 0.3	± 0.06	± 0.07	*(% wt.)*
Goldstein - Revised	± 6	± 4	± 1	± 1	*(% wt.)*
Lifshin - Generalized	± 5	± 4	± 1	± 1	*(% wt.)*

TABLE 4. Alloy 2 Homogeneity Level (3-Sigma Confidence)

	Al	Ti	Mn	Nb	
(Mean Concentration)	*(31)*	*(53)*	*(3)*	*(13)*	*(% wt.)*
Weight Percent Data Set	17%	7%	45%	9%	
Marinenko - ANOVA	19%	8%	45%	9%	
Goldstein - Original	1%	0.5%	2%	1%	
Goldstein - Revised	18%	8%	39%	9%	
Lifshin - Generalized	17%	7%	45%	9%	